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In situ Monitoring of Surface Oxide Reduction of Ru Nanoparticles in H_2 Flow as a Function of Temperature

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Beamline(s): X7B

Recently, an anode catalyst for hydrogen fuel cell made by spontaneous deposition of a submonolayer of Pt on Ru nanoparticles has been demonstrated to have a mass-specific activity for H_2 oxidation three times that of a Pt or Pt-Ru alloy. A key step in preparation of this catalyst is to heat the Ru nanoparticles in H_2 flow. A reduction of Ru surface oxide was proposed to occur so that the metallic Ru surface can act as the reduction agent for Pt spontaneous deposition. In order to verify this idea and to find the temperature range for a complete oxide reduction with minimized effect on the particle size, x-ray powder diffraction measurement was carried out at X7B.

The Ru/C (20%wt, from E-TEK) sample was loaded in a sapphire capillary. Under a constant H_2 flow, the temperature was increased at a constant rate from 25 to 800°C and H_2O in the exit gases was detected by a SRS residual gas analyzer. The time-resolved synchrotron x-ray diffraction data were collected by using a MAR345 area detector.

As shown in Fig. 1, the formation of H_2O was detected by an increase of H_2O signal around 100°C. Coincidentally, the diffraction intensity from metallic Ru nanoparticles increased sharply. These observations confirm the reduction of surface Ru oxide by hydrogen gas at temperature above 100°C. The width of the diffraction peak decreases significantly at t > 400°C, which suggests an increase of the Ru particle size. These results are consistent with the reactivity testing results for the samples prepared at different temperatures. Acknowledgments: Work supported by the US Department of Energy under Contract No. DE-AC02-98CH10886

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